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ANIMAL FATS IN HOT DIP TINNING

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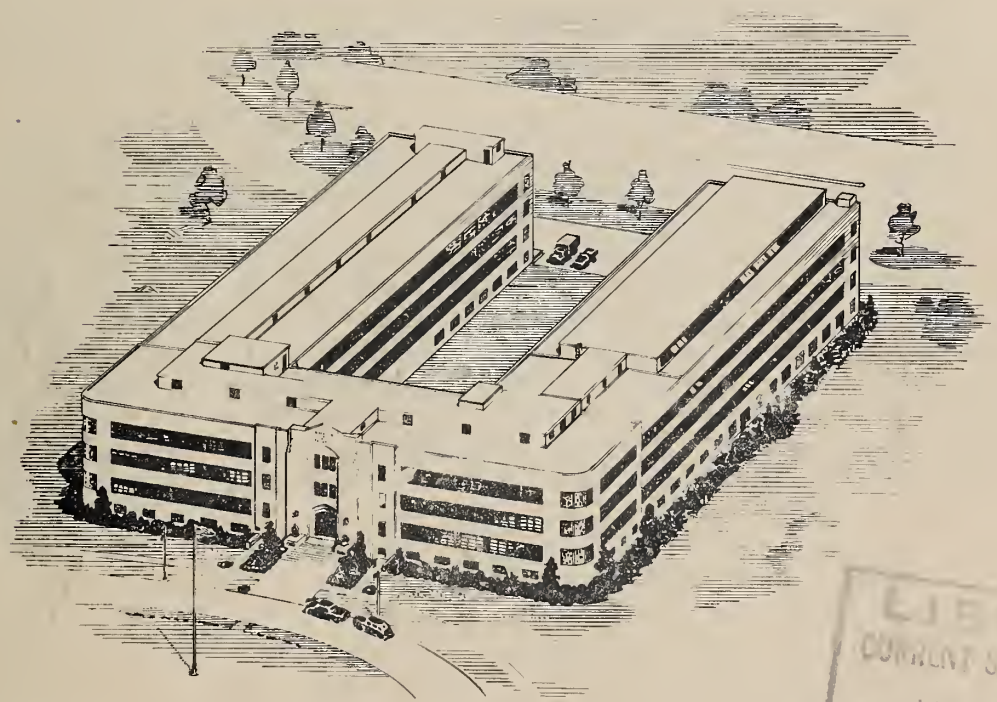

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FOREWORD

The development from present surplus domestic greases and tallows of a satisfactory replacement for palm oil in hot dip tinning would not only lessen our dependence upon imports of palm oil during national emergencies, but would also create a ready potential market for a surplus domestic commodity. About 15,000,000 pounds of palm oil are used annually for this purpose at the present time. The importance of the development appears even greater when it is considered that such replacement oils also hold a great deal of promise for satisfactory application to the cold reduction of steel sheets prior to tinplating.

The results of research reported herein show that typical grades of industrial fats, such as white grease, slightly modified by well known processing treatments, give satisfactory performance in both laboratory and mill scale tests for hot dip tinning. This work has been done by Armour Research Foundation in collaboration with the Eastern Regional Research Laboratory of the Bureau of Agricultural and Industrial Chemistry and in part, was performed under contract with the U. S. Department of Agriculture in accordance with authorization by the Research and Marketing Act of 1946. The contract was supervised by the Eastern Regional Research Laboratory of the Bureau of Agricultural and Industrial Chemistry. Work on this project was begun June 1, 1950, and was completed August 31, 1952; the final report being submitted November 20, 1952.

ANIMAL FATS IN HOT DIP TINNING[1]

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(1) A report of work done in part under contract with the U. S. Department of Agriculture and authorized by the Research and Marketing Act of 1946. The contract has been supervised by the Eastern Regional Research Laboratory of the Bureau of Agricultural and Industrial Chemistry.

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INTRODUCTION

During the development of the industrial process for tinning steel sheets by dipping them in molten tin, the addition of palm oil to the tin pot was found to be effective in removing tin oxides from the surface of the molten tin. This "fluxing" action is important because it makes possible the production of uniform, bright, tinned sheets. Palm oil has been used almost exclusively in this process ever since.

Until about a decade ago, there had been a few sporadic attempts made to introduce a domestic substitute for palm oil. These earlier attempts were unsuccessful owing partly to lack of knowledge of factors important for the satisfactory performance of oil in the tinning operation. Within the past ten years, however, laboratory investigations (1,2,3,4) have disclosed compositional and functional requirements for tinning oils. Armour Research Foundation had been engaged in this type of research sponsored by the American Iron and Steel Institute for two and a half years prior to the present investigation. They found that a carefully refined blend of deodorized tallow fractions containing a controlled amount of free fatty acids showed very promising performance characteristics for tinning. The present investigation may be considered a continuation of this study with special reference to the possibility of using ordinary commercial grades of greases and tallows suitably modified.

Eight different possible substitutes for palm oil, prepared from animal fats such as lard, grease, or tallow were given preliminary evaluation by laboratory tests designed to determine at least qualitative performance in tinning. Three of the substitutes were subjected to more extensive testing for certain properties, and one was selected for a plant scale hot dip tinning operation.

The results of these tests helped to establish the requirements and specifications for a satisfactory tinning oil and indicate that many of our most common grades of surplus animal fats can be successfully modified for this purpose. The mill scale operation, in which a partially hydrogenated, deodorized and stabilized white grease was employed, proved to be highly successful. The results are included in this report.

DISCUSSION OF HOT DIP TINNING OPERATION

The hot dip tinning operation consists of feeding sheets of pickled steel, by means of rolls and guides, through a large specially designed pot containing molten tin at about 600°F. The upper part of the pot is divided in such a way that the sheets are passed first through a flux which is floated on a section of the surface of the molten tin, then into the molten tin, and finally upward through an

18-inch thick layer of tinning oil which is maintained at about 465°F. A series of rolls which operate submerged in the oil smooth and reduce the thickness of the tin coating. As the tinned sheet leaves the oil it is cooled below the tin solidification point by air blasts. The sheet is then caught by other rolls which feed it to a wet washer and then to the branner, which removes excess oil by rubbing bran over the surface of the sheet. The tinned plate retains a very thin oil film on the surface; this film protects the plate and aids in fabrication operations.

REQUIREMENTS OF A HOT DIP TINNING OIL

Ference and coworkers (3) have listed the following characteristics for an ideal tinning oil:

1. It should make a smooth, bright, good quality tin plate.
2. It should drain rapidly and freely from the tinned sheet.
3. It should be of low volatility.
4. It should have high flash and fire points.
5. It should neither oxidize nor polymerize readily.
6. It should clean from the sheet readily in the washing and/or branning operation.
7. It should be of low viscosity at 100 to 210° F. so that it may be pumped or poured without difficulty.
8. It should be non-toxic and odor-free.
9. It should not become rancid during storage.
10. The film of oil remaining on the sheet should not interfere with lithographing or lacquering procedures, nor with soldering.
11. The film of oil should retard atmospheric corrosion of the sheet and aid in fabrication.
12. The oil should be readily available, preferably from domestic sources, and inexpensive.

Several disadvantages are inherent in the use of palm oil. The flash and fire point of new palm oil are quite low; losses due to volatility at tin pot temperatures are considerable; new palm oil must be conditioned by a preheat treatment to reduce danger of fire and to remove moisture before using; palm oil must be obtained from a foreign source which presents both strategic and economic problems from time to time.

Previous work (3) indicated that a carboxyl group was essential for proper drainage by the oil from the freshly tinned sheet as it emerges from the pot. This drainage property, frequently referred to as "dewetting" or "oil break", results in decreased oil consumption and may also be important from a plate quality standpoint.

LABORATORY EVALUATION OF TINNING OILS

In general, the procedure outlined by Ference et al (3) was followed for the laboratory evaluation of tinning oils. The viscosities were determined with a Zahn viscosimeter at temperatures of 210° F. and 465° F.

Eight samples of experimental tinning oils prepared from animal fats[a] by the Eastern Regional Research Laboratory were subjected to preliminary laboratory evaluation in order to select several of the most promising ones for more extensive study. These experimental samples are described in Table I.

TABLE I

Description of Samples of Experimental Tinning Oils

A. Prime Steam Lard, containing 0.1% phenylhydroquinone.
B. Filtrate Material (lard oil) from crystallization of prime steam lard from acetone (1 g. to 10 ml.) at 0° C., and containing 0.1% phenylhydroquinone.
C. Choice Tallow, deodorized.
D. Choice Tallow, deodorized, and containing 0.1% phenylhydroquinone.
E. Filtrate material from crystallization of choice tallow from acetone (1 g. to 10 ml.) at 23° C., and containing 0.1% phenylhydroquinone.
F. Choice White Grease, deodorized.
G. Choice White Grease, deodorized and containing 0.1% phenylhydroquinone.
H. Choice White Grease, refined, partially hydrogenated, deodorized, and containing 0.1% phenylhydroquinone.

NOTE: All the above samples were adjusted to contain from 7.5 to 8.5% free fatty acids.

(a) Some preliminary laboratory studies were made on two hydrogenated vegetable oils furnished by the Southern Regional Research Laboratory, but priority for edible uses made their extensive investigation at this time appear unattractive. Therefore, no data on these oils are included in this report.

The laboratory analytical and evaluation tests on these experimental samples are contained in Table II.

TABLE II

Laboratory Analytical and Evaluation Results for

Experimental Tinning Oils (Described in Table I)

Sample Designation	Iodine Value	Titer °C.	Flash Point °F.	Fire Point °F.	Activity* minutes	Odor	Heat Bodying Tests	Free Fatty Acids %	Polyunsat. Acid Content
A	65.7	39.0	490	550	33	S	S	8.6	12.5
B	78.0	31.8	435	585	40	S	S	7.6	15.3
C	47.0	42.5	475	560	28	S	S	8.0	3.2
D	47.0	42.5	460	550	68	S	S	8.0	3.2
E	50.8	40.7	490	565	67	S	S	8.6	3.6
F	63.6	39.0	480	565	50	S	S	7.7	10.6
G	63.6	39.0	470	565	33	S	S	7.7	10.6
H	52.0	42.5	500	655	31	S	Excellent	7.2	0.3

* Activity is determined by the petri dish test as described by Ference et al (3). The values indicate that all the samples had a satisfactory degree of activity.

S= Satisfactory

Viscosity increase with time of heating at hot dip tinning temperatures is considered to be a useful test in the selection of tinning oils. If the increase in viscosity is too great too much oil is dragged out with the tinned sheet as it emerges from the pot resulting in poor oil economy and more work in washing and branning. Laboratory tests without replenishment of any oil and without added flux were made to determine the viscosity increase with time of heating at 465° F. Viscosities were determined also at 210° F. at the same time intervals because the Zahn viscosimeter appeared to be more sensitive to viscosity changes at this temperature, and the measurements at this temperature are more directly related to the ease of cleaning of the tinned sheets.

The results of the laboratory testing for viscosity increase (Table III) indicate that all samples except Tallow[2] had as good or better resistance to viscosity increase (probably due to polymerization) than did palm oil. Samples G and H were particularly outstanding and showed low viscosities even after 12 days of heating. The data further indicate that phenylhydroquinone added as antioxidant also decreases the tendency of the fat to polymerize - as shown by less viscosity increase on heating. Samples A, B, D, E, G, and H (see Table I) contain added phenylhydroquinone. Samples F and G are the same except for the antioxidant; yet the latter showed much less increase in viscosity with time of heating. Sample H represents the same grease used in F and G except for partial and selective hydrogenation to reduce polyunsaturated components. This sample was superior to the others in viscosity character indicating that the content of polyunsaturated acids is an important factor in the increase of viscosity with heating.

On the basis of the results reported in Tables II and III, and for considerations of possible economy, Samples D, G and H were selected for more extensive study of the rate of viscosity increase in long term heating tests on 1500 g. of oil in the presence of molten tin in order to more closely simulate tin pot conditions. Viscosities were determined daily for up to thirty days, and amounts of oil were removed from the vessel to permit the addition of 18 percent of fresh oil daily. Under these conditions, an equilibrium viscosity is reached when little or no change in viscosity is noted over a three or four day period. Simple dip type viscosimeters made at Armour Research Foundation with internal dimensions much like that of the standard Saybolt tester were employed. An orifice diameter of 0.069 inches was used for measurements at 465° F. Samples D, G, and H had equilibrium viscosities of 45, 47, and 43 seconds, respectively, which compared favorably with 52 seconds for palm oil.

Reduction of the daily oil replenishment rate from 18 to 10 percent caused the viscosity of all the oils except Sample H to increase continuously over the period tested with no indication that an equilibrium viscosity would be achieved. Sample H, the hydrogenated white grease attained an equilibrium viscosity of 43 even at this low replenishment rate.

On the basis of the foregoing tests any of the three experimental samples would probably give performance equal to or superior to palm oil in hot dip tinning. The hydrogenated grease was selected for the mill scale tin plate production trial partly because the laboratory tests indicated it was superior to the other samples and also because it may provide a wider basis for selection of animal fats. Tallow had previously passed a mill test successfully.

SPECIFICATIONS FOR GREASES TO BE USED IN HOT DIP TINNING

The results of the laboratory experiments appeared to justify the following tentative specifications:

1. The starting material should be a Choice White Grease containing not more than 4 percent free fatty acids and not more than 1 percent moisture, insoluble impurities, and unsaponifiable matter. Low free fatty acid content is desirable to simplify the refining, hydrogenation, and deodorization treatments.

2. Alkali-refining of the grease is usually desirable to facilitate the hydrogenation. Extra high quality grease may not require this step or may require only a treatment with activated carbon and filtering.

3. The grease should be hydrogenated under selective conditions, such as 15 to 50 pounds hydrogen (gauge) pressures, temperatures of 140-160° C., agitator speeds of 400-500 r.p.m., and nickel catalyst concentrations of 0.1 percent. The hydrogenation should be controlled in such a manner that substantially only the polyunsaturated fatty acids will be reduced to monounsaturated ("oleic") acids. The product should be filtered at temperatures of 60-70° C. under conditions that remove the nickel effectively.

4. The filtered, partially-hydrogenated product should be steam deodorized at temperatures of 170-190° C. and pressures not to exceed 15 mm. of mercury, and for a length of time necessary to produce a bland product.

5. Phenyl hydroquinone or propyl gallate should be added to the deodorized product to the extent of at least 0.05 percent and mixed thoroughly at temperatures of about 60° C. to insure complete solution and dispersion.

6. The final product should be adjusted in free acid content so as to contain 8 to 9 percent. The fatty acids added should be very low in polyunsaturated acid content. Fatty acids obtained from selectively hydrogenated grease or tallow are satisfactory.

The final product should conform to the following specifications:

Free Fatty Acids	8 to 10 percent (calculated as oleic acid)
M. I. U.	1 percent maximum
Moisture	not greater than 0.3 percent
Color	not greater than 13 (F.A.C.)
Odor	bland
Titer	40 to 43° C.

TABLE III

Viscosity[1] Increase with Time of Heating at 465° F.

Days	0		4		8		10		12		16	
	210°F	465°F	210°F	465°F	210°F	465°F	210°F	465°F	210°F	465°F	210°F	465°F
Sample												
A-Table I	16	15	22	15	56	17	+	28	+	+	+	+
B-Table I	16	15	18	15	56	16	+	18	+	36	+	+
C-Table I	16	15	17	15	45	17	+	+	+	+	+	+
D-Table I	16	15	18	15	49	17	+	27	+	+	+	+
E-Table I	16	15	18	15	63	18	+	48	+	+	+	+
F-Table I	16	15	18	15	58	17	+	22	+	+	+	+
G-Table I	16	15	16	15	18	15	22	15	48	17	+	+
H-Table I	15	15	16	15	17	15	18	15	21	15	+	18
Palm Oil	16	15	22	15	50	17	+	+	+	+	+	+
Tallow[2]	16	15	23	15	+	+	+	+	+	+	+	+

[1] Expressed in seconds, G. E. Zahn Viscosimeter, #2 bore.

[2] Tallow investigated by Armour Research Foundation in previous work under the American Iron and Steel Institute sponsorship.

\+ Too great to measure.

Iodine Value	48 to 54
Total polyunsaturated fatty acids	not greater than 1 percent
Flash Point	470° F. or higher
Fire Point	570° F. or higher
Nickel Content	not to exceed 5 p.p.m.

PLANT EVALUATION OF EXPERIMENTAL OIL IN TIN PLATE MANUFACTURE

Before attempting a mill scale evaluation of the hydrogenated grease, a large quantity, about 7,000 pounds, had to be prepared for the purpose. This was prepared by Darling & Company, Chicago, Illinois, in conformity with the specifications furnished them for Sample H. This material was first analyzed and evaluated by laboratory tests prior to its delivery to Inland Steel Co., East Chicago, Indiana, for the mill test.

Preparation of Experimental Oil (Hydrogenated Grease): The starting material used in preparing the large quantity of Sample H was a choice white grease, the analyses of which are given in Table IV. This material was alkali refined and then hydrogenated at 17 p.s.i. and 255-300° F. The extent of hydrogenation desired is determined from the iodine value of the starting material and the amount of polyunsaturated acids present by calculating the lowering of iodine value required for reducing the polyunsaturated to monounsaturated acids. This lowering of the iodine value can be converted to its equivalent drop in refractive index for convenience in following the progress of hydrogenation. After hydrogenation, the material was filtered and deodorized by steam stripping at about 0.5" mercury pressure. The free fatty acid content was adjusted to about 9 percent, by addition of fatty acids from margarine stock and red oil (commercial oleic acid). Fatty acids from hydrogenated tallows or greases would ordinarily be added to bring the free acid content to 8-9 percent. However, if the hydrogenation proceeds somewhat beyond the calculated end point, the product may be too hard or high melting. In such instances, the product may be softened by adding oleic acid to make up the 8-9 percent free acid. The oleic acid should preferably be free of polyunsaturated acids. The final product should not have a titer greater than 42-43. Analyses of the processed grease is contained in Table IV.

This material was furnished for the mill scale tinning test. It is obvious that other grades of commercial fats and oils than white grease could be processed to yield a product similar to that of the hydrogenated grease and which would have similar analyses and properties. Particular attention should be given to keep the polyunsaturated acid content not greater than 1 percent.

TABLE IV

Analyses of White Grease Before and After Processing

	Before Processing	After Processing
Iodine Value	62.0	48.1
Titer	38.2°C.	42.3°C.
Free Fatty Acids (as % oleic)	3.4	8.5
Linoleic Acid *	6.9	0.8
Linolenic Acid *	1.0	0.2
Arachidonic Acid *	0.3	0.0
Oleic Acid *	50.6	51.2
Saturated Acids *	37.0	43.5
Flash Point (open cup)	--	520°F.
Fire Point (open cup)	--	655°F.
Odor on Heating to 465°F	Noticeable	Bland
Propyl Gallate (added)	--	0.1%

* Expressed as percent acids in sample.

Mill Operating Conditions: The operation of the tinning mill was left entirely to the trained plant personnel at Inland Steel Company. Records of operating temperature, tin coating weights, lacquer adherence, oil coating weight, staining properties, oil viscosity, free fatty acid content, oil consumption, bran consumption, production rate and quality of tin plate were maintained by personnel of the Research and Development Department of Inland Steel Company.

Production of tin plate during the period of the test was interrupted several times owing to labor difficulties occurring in the industry at the time. For this reason, the experimental tinning oil was subjected to a much more severe test than would normally be encountered in ordinary production runs. The over all time the oil was being heated to tinning temperatures (465° F.) compared to the actual time it was used for tin plating was much greater than that of normal operation, and may have influenced such values as viscosity and acid content.

A limited quantity of tin plate was produced first in a preliminary run using the experimental hydrogenated grease. This tin plate was then shipped to the American Can Company for organoleptic, fabrication, and corrosion tests. In all cases, the experimental plate was judged satisfactory. A special order was then placed by this company to cover the rest of the tin plate produced using the experimental oil.

Oil Viscosity: The oil viscosity was determined daily at the plant during the mill test and was reported in S. U. S. units at 210° F. The experimental oil had an initial viscosity of 57. During the period of the test the viscosity increased to as high as 224 before the addition of make-up oil. At the end of the test, the viscosity was 179. Palm oil operates very well at viscosities of 90-100 and is usually discarded when the viscosity reaches 120, because it exhibits poor "oil break" at this viscosity. In this respect, the experimental oil differed greatly from palm oil, in that no difficulties were experienced with "oil break" even at the high viscosities reported. Owing to the low oil consumption rate (see discussion below) the rate of replenishment of fresh oil to the tin pot was necessarily low. This low replenishment rate would be expected to give rise to higher viscosity oil in the tin pot at tinning temperatures because the average age of the oil in the pot becomes greater than that occurring with higher replenishment rates.

The oil viscosity determined daily during the mill test and reported in S. U. S. units at 210° F. were converted to their approximate equivalents of Armour Research Foundation readings at 465° F. This was done by taking periodic samples from the tin pot, determining their viscosities at 465° F. with the Armour Research Foundation viscosimeter and relating these values with those obtained as S. U. S. readings at 210° F. It is estimated that an equilibrium viscosity of about 75 seconds (ARF) would be established with continued operation.

Oil Consumption: The mill personnel were aware that palm oil is discarded when its viscosity exceeds 120 S. U. S. at 210° F. because it exhibits poor oil break at this viscosity. This limitation was not applied to the hydrogenated white grease owing to its good oil break character at even greater viscosities.

Oil was added to the tin pot, one barrel at a time as needed, during the test. The replenishment rate was about one-third that required for palm oil. The consumption of the experimental tinning oil based on the production of almost 30,000 base boxes of tin plate was 0.10 lbs. per base box, compared to the established average of about 0.32 lbs. per base box for palm oil. (A base box is the tin plate industry's unit of area measurement and is equal to 31,360 square inches of tin plate. Since the tin plate has two tinned surfaces, a base box has 62,720 square inches of tinned surface.)

Free Fatty Acid Content: The tinning oil, as charged to the tin pot, contained about 8.5 percent free fatty acid. The acid content dropped rapidly during the first few hours of heating and had reached 5.3 percent after 72 hours on heat. In the first nine days of mill operation the acid content gradually dropped to 3.8 percent, following which it gradually increased and at the end of the run was about 7.7 percent.

The laboratory experimentation on the formation of free fatty acids would seem to indicate that the low acid content was caused by the low tin plate production rate during the initial portion of the test. (This low production rate was the result of labor difficulties and not technical difficulties.)

It is difficult to predict accurately the final free acid content had the use of this oil been continued indefinitely, however, it is believed that an equilibrium acid content of 6 ± 2 percent would be established.

Volatility and Odor: Although no quantitative measure of the fraction of the oil lost by volatility was possible, the higher flash point, together with the observably less dense smoke from the pot, suggests that a part of the improved consumption value obtained was due to a smaller proportion being lost up the ventilating stack. A slightly adverse report was obtained on the odor of the hot oil which was described as being "sharp-pungent". This report is believed to be largely due to the fact that the test set-up was such that the reserve storage tank contributed materially to the total vapors in the area at a location where ventilation was less than ideal. It seems probable that this condition could be easily rectified under normal operating conditions with a closed oil recirculating system and/or with improved ventilation.

EVALUATION OF EXPERIMENTAL TIN PLATE

Of the nearly 30,000 base boxes of tin plate produced during the plant test, about 98.0 percent was prime tin plate; "menders" amounted to 7.5 percent and "wasters" amounted to 1.5 percent. "Menders" are plates that do not have a uniform tin coating and must be put through the tinning mill a second time. "Wasters" are caused by steel defects or handling accidents and are considered as scrap.

Analysis of the menders indicated that the major portion of these rejects occured on start-up shifts and for a few shifts later.

The normal amount of menders is 6 percent with 4 percent wasters. It should be noted, however, that many of the wasters are the result of handling and have little to do with the functioning of the oil. Since this was an experimental test, the operation of this tin pot may have been more carefully attended resulting in fewer wasters.

Mechanical condition of the tin coating machine also has a marked effect upon the quality of tin plate produced. The plate produced during the test was "B" type plate which has an aim of

1.00 lbs. of tin per base box. The coating weight was determined twice during each production shift (8 hours per shift). During the first portion of the test, the variation in tin coating weight was slightly normal, however, not seriously so. Replacement of the tinning machine improved the uniformity of coating weights and the variation in coating weight was then in the range experienced with palm oil.

Lacquer Adherence: Some 850 samples of tin plate were tested for lacquer adherence, using phenolic lacquer, by the American Can Company method. All samples were given the rating of O.K.; no samples were rated with an average grade of fair or poor.

Stain Test: Samples of production run sheets were baked for 30 minutes at 410° F. No sheets showed any yellow or brown stains.

Porosity: Porosity of the tin plate, as determined by the Ferrocyanide method, indicated that the porosity of the plate was substantially in the same range as when processed with palm oil.

Oil Coatings: Oil coating of the finished tin plate was 0.39 grams per base box. This is similar to the aim of normal production of hot dip plate using palm oil.

Can Company Evaluations: Four can companies were supplied with samples of tin plate produced during the test; their evaluation are of utmost importance in the consideration of the potentialities of the experimental oil in hot dip tinning. Summarizing statements taken from their reports are as follows:

1. Organoleptic tests on samples of the oil and on containers fabricated from the experimental tin plate were satisfactory.

2. The tin oxidation rate, as determined by an accelerated aging test on the experimental plate, was equivalent to the rate on regular production plate.

3. A semi-commercial enameling and fabrication test was conducted using the experimental plate. Some of the plates had been lithographed and enameled before fabrication. The plate performance was satisfactory.

4. Adhesion, tackiness tests, and process evaluation of modified phenolics, "C" enamel and "R" enamel, were equal to the results obtained with palm oil plate.

5. In both laboratory and factory evaluation of the experimental plate, its general acceptability justifies further trials on a commercial basis.

COST ESTIMATES OF EXPERIMENTAL TINNING OIL

The experimental oil was made from choice white grease by refining, hydrogenation, deodorization, and blending with fatty acids up to 8 percent free acids. Antioxidant was added to the extent of 0.1 percent. It is estimated that all this treatment would add from 2 to 3 cents per pound to the initial cost of the grease without allowance for other costs, such as depreciation of equipment, sales commissions, etc.

It appears, therefore, that the processed grease may well be less expensive than tallow or palm oil, particularly when the low consumption rate of the grease, as indicated by the mill test, is taken into account.

CONCLUSION

It is concluded that the better grades of greases and tallows with appropriate processing perform as well or better than palm oil in hot dip tinning. The cost of the processing is not prohibitive and the price of the finished oil is favorable compared to average prices of palm oil.

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The mill trial of the experimental oil was conducted by the Inland Steel Company, Ross L. Harbaugh, Chief of the Chemical Laboratories, Research and Development Department, and E. D. Martin, Superintendent of the Research and Development Department, supervised the tests while J. E. Joyce and I. C. Katsahnias recorded pertinent data and prepared the test report.

The preliminary organoleptic, fabrication, and corrosion tests on the tin plate were conducted by the American Can Company under the supervision of Dr. L. P. Gotsch. The evaluation of the experimental tin plate was under the direction of W. S. Irvine, Supervisor, Metallurgical Group, Central Division Laboratory.

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